

LOW SOLAR ABSORPTANCE AND EMITTANCE
SURFACES FOR MARS ENTRY CAPSULE
AND NEAR SOLAR MISSIONS

NAS 2-3063

QUARTERLY PROGRESS REPORT FOR PERIOD
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FOREWORD

This report was prepared by the Thermophysics Section of the Aerospace Sciences Laboratory, Lockheed Missiles & Space Company (LMSC) for the National Aeronautics and Space Administration, Ames Research Center, Moffett Field, California, under contract NAS 2-3063. This report summarizes the progress on the contract for the first quarterly period from 29 June 1965 to 15 October 1965.

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ABSTRACT

The objective of this program as stated in Reference 1 is to investigate techniques and material for finishing spacecraft structural surfaces with reproducible, stable, thermal control materials. Emphasis will be placed upon achieving solar absorptance (α) values between 0.03 to 0.10 and total hemispherical emittance (ϵ) values at room temperature between 0.02 and 0.85. Specific goals will be surfaces with (a) α/ϵ ratio less than 0.1, (b) α/ϵ ratio \approx 1.0 with α less than 0.1 and (c) minimum emittance. The resulting surfaces will be evaluated for use on spacecraft operating as close as 0.2 AU and at distances of 2.0 AU.

This report summarizes the progress made during the first quarterly period of the program. Included herein are the program description and sections on sample preparation, material description, equipment and apparatus, experimental procedures, experimental results, and future work.

Section 1

INTRODUCTION

This program involves investigations of several coatings with various substrates. Environmental stability studies and optical property measurements are to be performed. Vapor deposition equipment is to be assembled with major apparatus items to be purchased. The program is described below under the headings of coating systems, substrate requirements, environmental stability studies, measurements, and purchased apparatus.

Coating Systems

1. Experimental methods of producing practical surface systems with low α_s , low ϵ and low ratios of α_s/ϵ_H varying from 2.0 to 0.07 utilizing transparent high emittance coatings will be investigated. Descriptions of the application techniques including purity of material, deposition, rate, pressure, thickness, and substrate temperature will be made.
 - a. Specific metals to be studied will be the following:
aluminum, silver, and gold.
 - b. Specific dielectric materials to be studied will be the following: silicon oxide (vacuum deposited), aluminum oxide (vacuum deposited), and high-purity quartz.
 - c. The following surface systems are to be investigated:
 - (1) α_s/ϵ ratio less than 0.1: OSR-TP-060.
 - (2) α_s/ϵ ratio ≈ 1.0 with α_s less than 0.1: silver; silicon oxide.
 - (3) Minimum emittance, aluminum, silver, gold, silicon, oxide, aluminum oxide.

- d. Fifteen samples of each of the final three coating systems selected for environmental exposure will be prepared on 15/16 inch diameter 6061-T6 aluminum substrates.

Substrate Requirements

1. Substrate materials will be limited to the following: one selected aluminum alloy, one selected stainless steel, and one form of quartz.
2. Experimental studies will be performed with flat surfaces. In addition, the OSR-TP-060 studies shall be performed with high L/D cylinders with nominal diameters of 1 inch.
3. Evaluation of substrate preparation and polishing requirements will be performed in terms of compatibility with the candidate coating system.

Environmental Stability Studies

The specimens are to be exposed to simulated solar ultraviolet (U.V.) radiation. Sample irradiances will be at least 10 "suns" (intensity between 0.2 and 0.35 microns) under high vacuum conditions (less than 1×10^{-6} torr) using ion pump equipment. All temperatures quoted are $\pm 25^{\circ}\text{F}$. The following tests shall be performed:

1. Screening tests with approximately 10 "sun" U.V. at $500^{\circ}\text{F} \pm 25^{\circ}\text{F}$ for 250 sun-hours and thermal cycling from 70°F to $+300^{\circ}\text{F}$ ten (10) times.
2. Final tests on selected systems (not to exceed 3) shall be performed for 2,000 sun-hours at temperatures of 70°F and 500°F .

Measurements

1. Measurements of the normal spectral reflectance from 0.27 to 19 microns for the systems selected for final tests (maximum of three) will be performed before and after exposure.
2. The α_s/ϵ ratio will be measured for the systems selected for the final tests. The α_s/ϵ ratio will be measured at the equilibrium specimen temperature with normal incident radiation of one "sun" flux density.

3. Solar absorptance (α_s) will be calculated based upon normal spectral reflectance data and the most recent solar spectrum data available.
4. Total hemispherical emittance will be measured as a function of temperature over the range of 70°F to +500°F for systems selected for exposure to final environmental tests. Total hemispherical emittance will be calculated for these specimens at 70°F, 140°F, 240, 340°F, and 540°F from reflectance data and measured at room temperature with the Lion Emissometer.

Purchased Apparatus (Major Items)

1. Electron beam evaporator
2. Electron beam power supply
3. Thin film thickness measurement and monitoring unit.

Section 2

SAMPLE PREPARATION

The Optical Solar Reflector (OSR) will be made with fused silica. A thickness of 6 to 8 mils will be used for flat surfaces. This should provide an emittance within approximately 2% of the maximum achievable value.

The measurement of total hemispherical emittance of the OSR with a cylindrical calorimeter will be performed with commercially available thin wall fused silica (28 to 51 mils).

The experimental studies with high L/D ratio cylinders will be performed with fused silica. Preliminary studies of coating techniques may be performed with Pyrex tubing or cylinders. A nominal ratio of 6 to 1 will comprise the high L/D ratio studies. The Optical Solar Reflector in all cases will be applied to 6061-T6 aluminum substrates.

Section 3

MATERIAL DESCRIPTION

Vacuum Deposition Source Material

The source metals to be evaporated for low solar absorptance and/or emittance are aluminum, silver, and gold. The dielectric overlays for controlling emittance are to be silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3).

The material form, purity levels, and source are as follows:

Aluminum

99.9998% purity

Zone refined

Cominco 69, pellets

Impurities, ppm by weight

Ca	0.1
Cu	0.1
Mg	0.2
Mn	0.2

Silver

99.9999% purity

Vacuum Melted (electrolyzed)

Cominco 69, shot

Impurities, ppm by weight

Bi	0.1	Mg	0.1
Ca	0.1	Si	0.1
Cu	0.1	O_2	2.1
Pb	0.1		

Gold

99.9999% purity
Zone refined
Cominco 69, shot

Alumina

Sapphire Boules
Linde-Union Carbide

Impurities in material to grow crystals, ppm by weight

Mg	2.0	Ga	10
Si	30-60	Cu	< 1
Ca	10-20	Ag	< 1
Fe	2-5	Cr	Trace
Pb	50-100	Ni	Trace

Estimated boule purity greater than 99.99999%

Silica

Quartz chips
Suprasil
Englehard Industries
1 ppm impurity or less.

Substrates

The substrate materials are Aluminum Alloy and stainless steel. For controls, glass and quartz substrates will be used for surface quality reference.

Aluminum Alloy

6061-T6 (1/32" thick)
Condition A mill finish
Condition B mirror polish

Stainless Steel

Type 430

Republic Steel #2

Bright annealed

condition:

mill finish

(mirror surface)

Glass

Corning #7059

(low alkali, less than 0.2 percent)

Barium aluminum silicate.

Fusion formed surface.

Typical surface smoothness is 60 angstroms.

Quartz

Optically ground and polished

Esco Type C0

OSR-TP-060

Adhesives and cements are being procured for evaluation for use with OSR-TP-060. Of special interest and concern is the requirement for service to 500°F. Experimental quantities of the following adhesives, primers, and cements have been obtained.

1. Dow Corning 93-025 and catalyst (adhesive)
2. Dow Corning 93-033 and catalyst (adhesive)
3. Dow Corning 92-018 (adhesive, one-part)
4. Dow Corning 92-022 (adhesive, one-part)
5. Dow Corning 92-033 (primer)
6. Dow Corning RTV 1200 (primer)
7. General Electric RTV 615 and catalyst (adhesive)
8. General Electric "Metal Seal", metal-colored silicone rubber
(adhesive, one-part)
9. LMSC-LP40A, silicate (cement)
10. Philadelphia Quartz Kasil 88 (cement)

Section 4

EQUIPMENT AND APPARATUS

The equipment consists of a basic vacuum system, electron beam multiple source evaporator and power supply, evaporation rate monitor and controller, ion bombardment system, and evaporation chamber.

Vacuum System

An existing 6-inch vacuum system of a nominal 1500 liters/second capacity with a large capacity anti-creep liquid nitrogen trap is to be used. The system has been modified with a high vacuum by-pass valve to permit control of gas pumping during ion bombardment. The system is presently complete and operational.

Electron Beam Unit

The electron beam unit consists of a commercial power supply and gun-crucible assembly.

The power supply (Temescal/EBH-5M3) is rated at 10 KV and 1/2 amp and has a controllable constant power output. The power level can be controlled manually or automatically in conjunction with the evaporation rate controller.

The electron beam gun (Temescal 10 LTFH400) consists of an electron emitter and a four position water cooled copper crucible, externally positioned, enabling multiple material evaporations during processing.

In order to evaporate aluminum at the desired high rates, ceramic crucibles will be inserted in the water-cooled copper cavities. These act as a thermal resistance and also prevent wetting of the aluminum to copper. The ceramic crucible is a National Carbon "intermetallic composite" (primarily titanium diboride) which was developed for aluminum evaporation. It does not react with aluminum and is oxidation resistant. To lessen the chance of the dissociation of the crucible material at the high evaporation temperature used with aluminum (1800-2000°C), high density alumina crucible liners have been fabricated. These have less thermal shock resistance than the crucibles, but will minimize crucible dissociation impurity condensation on the substrates.

The electron beam supply and gun assembly have been installed but not checked-out. Building service has been connected to the power supply.

Evaporation Rate Monitor

A Sloan rate monitor and controller (OMNI) has been received. The quartz transducer assembly is being mounted on the substrate holder.

Operational checkout of this unit is pending the completion of the evaporation system.

Ion Bombardment System

An existing high voltage, high reactance supply will be used to induce ionization.

An ionization cathode, which is part of the source shutter assembly, has been fabricated.

This phase of fabrication is complete.

Evaporation Chamber

The evaporation chamber includes a ground stainless steel box, a 14" pyrex cylinder, and a top flange to which the substrate holder is attached.

The steel chamber contains the evaporator, ion bombardment electrode and source shutter assembly, high voltage and water circuit lead-thrus.

The substrate holder provides eight substrate positions. Each position is independently shuttered. The shutters are actuated with bi-directional motors, remotely controlled, which enable depositions of varying film thicknesses while maintaining deposition parameters constant.

Approximately 90% of the assembly is complete. Fabrication remaining to be done is shutters, mounting of monitor transducer, substrate holder, and source shutter.

Section 5

EXPERIMENTAL PROCEDURES

To evaluate the adhesives to be used with OSR-TP-060 preliminary screening tests are being performed. These consist of applying OSR-TP-060 segments to 6061-T6 aluminum substrates. Flat substrates are used, but the suitability of the adhesive for application to one inch cylinders is observed. The cured specimens are inspected for corrosion. They are heated to 500°F and thermally cycled between 500°F and room temperature. These preliminary thermal cycling tests are performed at atmospheric pressure. Thermal cycling tests with the more promising candidate adhesives will be performed in a vacuum, and the final environmental stability tests will include ultraviolet radiation. The equipment and apparatus for the environmental studies are described in Reference 2.

Section 6

EXPERIMENTAL RESULTS

When tested as cements for use with OSR-TP-060, LP40A and Kasil 88 proved to be noncorrosive and to have good adhesion. However, the lack of elasticity and poor cohesive strength resulted in failures due to thermal stresses under thermal cycling. The preliminary tests indicate these inorganic cements to be unsatisfactory for use in this program.

The one-part adhesives, Dow Corning 92-018, Dow Corning 92-022, and General Electric "Metal Seal," caused no corrosion. Their high viscosity would make them suitable for application to cylinders, but techniques would have to be devised to apply a thin layer of uniform thickness to cylinders and flat surfaces. However, some separation occurred at the bonding surfaces of the OSR upon heating. This separation is apparently due to non-uniform dimensional changes in the silicone (probably related to shrinkage on curing). For Dow Corning 92-018 and General Electric, "Metal Seal," the separation occurred at approximately 300°F. The separation occurred at an indicated furnace temperature of 440°F for Dow Corning 92-022. Curing the one-part adhesives does not seem to be a problem, but their performance at elevated temperatures is apparently marginal at best.

Section 7

FUTURE WORK

During the next quarter:

1. Complete system fabrication and assembly.
2. Inter-connect system with power supplies and controllers.
3. Mount high voltage isolation, high current filament transformer near chamber and enclose for personnel protection.
4. Check-out components and component assemblies for operational integrity.
5. Make trial runs for checkout of system functions and modify as required.
6. Make trial evaporations to permit calibration of rate monitor and controller, by interferometric means, using various source materials.
7. Continue evaluation of adhesive for use with OSR-TP-060 to 500°F.
8. Study the problem of covering one-inch cylinders with OSR-TP-060 using commercially available materials.

Section 8

REFERENCES

1. Contract NAS 2-3063, "Low Solar Absorptance & Emittance Surfaces for Mars Entry Capsule & Near Solar Missions," effective date 29 June 1965.
2. Lockheed Missiles and Space Company, Proposal for Studies of Optical Solar Reflectors for Thermal Control of Solar Probes, LMSC-893893, Sunnyvale, California, 2 April 1965.